

AIAA-94-3241 Developments in Fullerene Ion Propulsion Research

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DEVELOPMENTS IN FULLERENE ION PROPULSION RESEARCH

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Abstract

Several reports of successful operation of DC discharges using fullerene vapor have been made. However, degradation of the propellant molecules in the vapor source and in contact with the filament cathode was observed in each case. Because disintegration of the fullerene cage could result in poor engine performance. or thruster failure, we have developed an RF ion thruster for use with C60 propellant to avoid the presence of electrodes inside the discharge chamber. Also, we have conducted a series of experiments to ascertain the stability of fullcrenes at high temperatures. We find that when C60 and C70 are maintained at temperatures above 1073 K, or arc exposed 10 hotmetal surfaces, significant degradation occurs. We find an activation energy of 261.5 kJ/molfor this process. At temperatures below 873 K, no breakdown of fullcrenes can be detected, even after heating for 6 hours. The effect of these findings on C60 ion thruster design is discussed.

Background

Since 1991, three groups 1-3 have reported on the development of an ion thruster which utilizes the all-carbon mole.culc.s known as fullerenes as a propellant. Discussions of the potential performance advantages of fullerene fueled ion propulsion have been published 4,5. Anderson and Fitzgerald,3 and Hruby cl al. both successfully sustained DC fullerene plasma discharges using thoriated tungsten filament cathode ion sources. Anderson and Fitzgerald confirmed the presence of fullerene ions by mass spectral analysis of the extracted ion beam. Hruby et al. detected fullerene material deposited on optical surfaces using Fourier Transform Infrared (FTIR) spectroscopy. Both of the sc groups reported substantial erosion of the filament cathodes used in their devices. The presence of a significant quantity of toluene insoluble carbonaceous material was observe.d in the effusion cell by both groups after healing.

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Takegahara and Nakayama² have reported an unsuccessful attempt to establish an RF generated plasma using fullcrenes. They found that their quartz discharge chamber wall temperature was too low, malting in condensation of the fullcrene propellant. Takegahara and Nakayama obtained FTIR absorption spectra of original and condensed C60 powder. If normalized, their spectra indicate a J0ss of material similar to that observed by Hruby c1 al. ¹ and Anderson and Fitzgerald.³

RF Plasma Discharge Experiment

To avoid the presence of high temperature metal surfaces other than the extraction grids, we have constructed an RF discharge chamber similar to that of Takegahara and Nakayama². We are using an Eni Power Systems 13.56 MHz RF power generator (model 600A) and impedance matching circuit (RF Services) to reduce reflective RF 10sscs from the engine. A schematic of the RF thruster assembly is depicted in Figure 1,

The discharge chamber consists of a 7.0 cm high, 7.0 cm diameter cylindrical quartz vessel with an extraction grid system and an oven containing the fullerene propellant flanged to opposite ends. An RF coil wrapped around the outside of the quartz vessel induces an azimuthal electric AC field inside, the chamber. The fullerene plasma is maintained inductively, climinating the need for hot electrode surfaces inside the discharge chamber, ignition of the discharge may be obtained by RF breakdown or with the aid of electrons attracted from the neutral izer filament into the discharge chamber by temporarily applying a positive voltage to the screen grid. The RF field further accelerates the electrons inside the discharge chamber, leading to breakdown. Because of the difficulties encountered by Takegahara and Nakayama² in obtaining high wall temperatures, we are investigating the use of graphite vam around the quartz vessel which can be both inductively heated and resistively heated by an independent power supply.

A small orifice in the side of the effusive cell faces an Inficon model XTM/2 Quartz Crystal Microbalance (QCM) 10 give real-time flow rate measurements. Prior attempts 10 monitor flow rate by forming a capacitor wi(h the effusive cell and a C_{60} dielectric failed because the capacitance change due to thermal effects were of the same order of magnitude as the change due to loss of the C_{60} dielectric.

The QCM sensor is placed 0.27 m from the quartz effusive cell nozzle. Calibration is performed by monitoring the deposition rate in ng/sec. The fullerene sample is weighed before and after hc.sling to determine the. total mass of evaporated material.

Beam diagnostics have thus far been limited to the use of an EXB mass spectrometer which displays peaks of all of the species present in the ion beam. Resolution of large mass ions in this device is not high enough to distinguish between the various fullerenes. 'I'his was verified during DC discharge experiments when two peaks corresponding 10 singly charged and doubly charged fullerenes were observed.³ No attempt to detect the presence of neutral species was made. Modifications to the EXB probe are underway to improve resolution in the high mass range. We

have also obtained a Beckman IR-18A infrared spectrophotometer which we intend to use for ion beam analysis. The instrument is sensitive in the 400010600 cm⁻¹ range.

The RF ion engine was operated using xenon as the source gas. The thruster was equipped with an optics system consisting of stainless steel screen and accelerator grids. The grids were spaced 1.6X 10³ m apart and had a set of 19 matching 3.2 X 10-3 m diameter holes. They were operated at a total-acceleration voltage of 3.2 kV and a net-accelerating voltage of 1.9 kV.

The discharge could be initiated at an RF power level of SO W; subsequently, the power could be reduced below 10 W before. the plasma would extinguish. Below 40 W, the discharge appeared very dim and no current was extracted from the optics system. Above 40 W, the discharge became considerably brighter and ions were extracted. Figure 2 shows the extracted beam current as a function of RF power when the ion engine was operated at a xenon flow rate of 0.094 mg/s. The beam current increased rapidly with power between 40 and 100 W, then leveled out to a maximum of 2.9 mA.

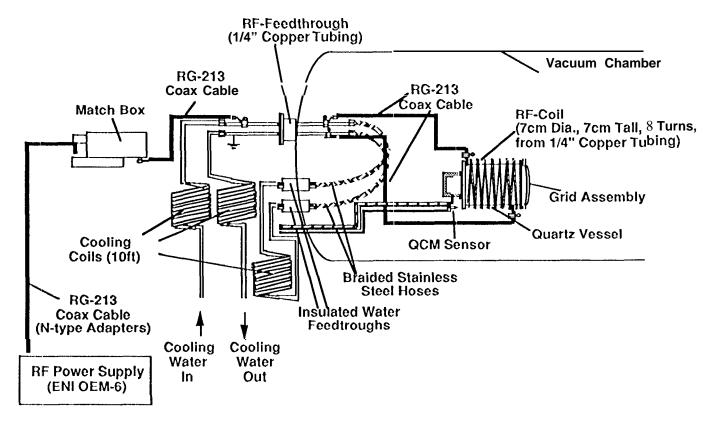


Figure 1. Schematic of the RF thruster assembly.

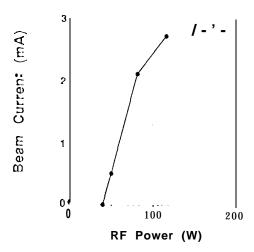


Figure 2. Beam current as a function of RF power for xenon plasma discharge,

Thermal stability and material compatibility of C₆₀ and C₇₀

"1 'here have been numerous studies of the oxidative behavior of C60 and C70⁶⁻¹1 conducted since fullerenes became available in macroscopic quantities in 1991. Iixperimental and theoretical 13 investigations of thermal decomposition of fullerenes have also been reported.

Sundar et al, ¹² sealed pellets of ^C60 in evacuated ampoules and heated them to temperatures between **773** K and 1173 K for 24 hours. X-ray diffraction and IJV-visible absorption spectra of the samples showed increasing degradation with higher temperature. However, adequate preheating of the samples under dynamic. vacuum to remove solvents and adsorbed impurities was not performed, nor has such a study been conducted where the decomposition in vacuum was evaluated as a function of time.

The reactivity of C_{60} with metals is of interest 10 the high temperature superconductor community. Alkali-metal-doped fullerenes have the highest known superconducting transition temperatures for organic superconductors. I'bus, the interaction of C_{60} with alkali metals at temperatures below 500 K has been investigated $^{14,1^{\rm s}}$ in attempts to form compounds.

'1'here arc several possible mechanisms for C60 degradation. These arc 1) thermal disintegration (vibrational modes excited beyond bond-strength limitations), 2.) stoichiometric

reaction with impurities, 3) catalysis with impurities, 4) catalysis with container walls, 5) catalysis with amorphous carbon fragments, or 6) a combination of any of the preceding mechanisms.

11.1.. Zhang et al.] ³have. reported molecular dynamics simulations modeling the process of thermal disintegration of fullerenes. They predict fragmentation temperature as a function of cluster size for fullerenes containing 20 to 90 atoms. Not surprisingly, C60 and C70 were. shown 10 be more thermally stable than other fullerenes. It was also found that the disintegration temperature became reasonably constant for molecules containing more than 60 carbon atoms. Zhang et al. interpreted the fragmentation temperature as a measure of the weakest bond in the mole.cric. Hence, their fragmentation temperature corresponds to the onset of bond-breaking in the fullerene cage.

The predictions of Zhang et al. ¹³ agree quite well with measurements of the heat of formation of C60 reported by References 16-18 to be 2.30X 10³ kJ/mol, 2,28X 10³ kJ/mol, and 2.42 X 103 kJ/moles, respectively. Using the interpretation of Zhang et al. of thermal disintegration, we find a lower limit of an Arrhenius activation energy for the process to be 38.3 kJ/mol, corresponding to a temperature of 4610 K.

A.M. Vassallo et al. ⁷ performed an emission FTIR study of solid-slate C₆₀ and determined thermal stability of the material at the highest temperature of their experiment, 873 K, Significant sublimation of the fullerene material appears to have made the experiment impractical at higher temperatures. When a 25% oxygen in argon mixture was introduced to the C₆₀ sample during heating, all C₆₀ emission peaks were seen to disappear by 723 K. C02 and CO emission bands were observed, as well as features from reaction products that may be attributed to cyclic anhydrides.

Hruby et al. ¹carried out tests of fullerene material compatibility with stainless steel, molybdenum, alumina, boron nitride, aluminum nitride, and quartz. They reported that no reaction of the fullerenes occurred with any of these materials, but chose quartz for their discharge chamber with molybdenum and stainless steels grids. Anderson and Fitzgerald³ used both graphite and stainless steel sources and grids. In both sources, notable degradation of the propellant molecules at high temperature was confirmed by FTIR spectroscopic analysis of the powder remaining in the effusive cell and on the walls of the discharge chamber. However, mass spectral analysis of the ion beam did not indicate the presence of either C₂ or C₄ fragments which would be expected from dissociated fullerenes. ¹¹11 was also

found that removal of residual solvents and impurities by heating the mixed fullerene samples under dynamic vacuum at 473 K for 12. hours prior to operation of the discharge did not inhibit the fullerene breakdown.

Thoughnot exploring any particular application, 1).11. Yuet al. ¹⁹ investigated the formation and fragmentation of fullerene ions in an electron-impact ion source. Unimolecular dissociation rate constants for the process of fragmentation via removal of even-numbered carbon fragments were obtained.

Because propellant flow rate is determined by the. fullerene vapor source temperature, knowledge of the reactivity and stability of C60 and C70 molecules al high temperatures is important to fullcrene ion thruster development. Excessive degradation will impact the total propellant utilization efficiency, as well as increase the possibility of thruster failure duc to the presence of condensed fragments and reaction products. The fullerene sources may require heating to over **800** K for adequate fullerenc pressures to be obtained. Figure 3 shows experimentally-determined vapor pressure curves for C60²⁰⁻²³. Hollow cathode operation with fullerene vapor for either DC plasma generation or ion beam neutralization would require that C60 be stable at temperatures over 1300K. 11 is apparent that an in-depth look at the high temperature behavior of fullerenes is required,

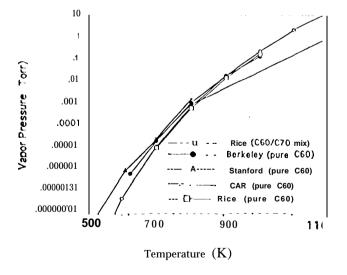


Figure 3. Vapor pressure as a function of temperature for C_{60} and C_{60}/C_{70} mix $^{20-23}$.

Experiment

We have performed detailed fullerene thermal and material compatibility tests to determine the source of residue in fullerene ion thruster experiments. The tests were performed by placing approximate.ly 10 mg of fullerene powder in quartzampoules cleaned in acetone and freon. We pre.pared sets of 5 ampoules at a time, containing either pure C60 obtained from MER Corporation, pure C70 from Term I.TD., or a fullerene mix composed of approximately 85% C60, and 15% C70. The fullerene mix was Soxhlet extracted with toluene from soot produced by Ulvick Industries.

The ampoules were placed under vacuum in the low 10⁻⁶ Torr range. Partial pressures of monatomic and diatomic oxygen, CO, C02, 011, }120, toluene, and benzene were monitored using a calibrated MKS model 600A PPT Residual Gas Analyzer (RGA). Mass spectra of all contaminants (under 100 amu) in the vacuum system were also obtained.

The fullerene samples were heated to 473 K for 3 hours, then to 523 K for an additional Ito 3 hours, until the partial pressure of toluene fell below 2x10-8 Torr. in all cases, the re was a rapid and significant increase in toluene partial pressure, with moderate increases in O2 and H2O.C60 epoxide may have been another source of the observed oxygen. In the case of the C70 samples, both C02 and CO partial pressures increased more rapidly than they did during either the pure C60 or mixed fullerene sample heating. The total pressure in the vacuum system subsequently increased beyond 2x10-4 Torr (the upper limit of the range of the RGA) for several minutes after the ampoule heaters had reached steady-state temperatures.

The anneal temperature we used for solvent removal from the fullerenes in the quartz ampoules was between 473 K and 523 K. Although this temperature is high enough to remove a significant portion of adsorbed water on the surface of the ampoule, it is insufficient for removal of the hydroxyl groups terminating the quartz surface, It is necessary to heat the quartz under dynamic vacuum to over 873 K to remove these surface impurities in the form of 120 and 112.24 To determine whether these silts on the quartz surface were serving as catalysts for the fullerene decomposition, we formed an ampoule with a "pocket" for holding the fullerene test sample while the bottom portion of the ampoule was heated with an acetylene torch. The remainder of the experiment was performed as before.

The ampoules were then vacuum scaled and placed in a tube furnace for durations of 20 minutes, 1 hour, 3 hours and 6 hours. **Wc** prepared sets of samples heated to 873 K, 973 K,

1073 K., 1173 K, 1273 K, and 1473 K. One trial consisted of ampoules that had been backfilled with approximate.ly 5 Torr of Ilelium.

For total elimination of toluene impurities, Milliken et al. 6 found that healing of C60 to 483 K for 16 hours was sufficient. To verify that the four to six hour bake was adequate to drive off contaminants, we heated five samples of (he. fullerene mix under dynamic vacuum for 16 hours at 473 K and compared these samples with [hose heated for shorter durations.

'1"O study the effect of hulk metals in contact with fullerenes at high temperatures, we placed 5.0 cm long by 0.32 cm wide metal strips in the ampoules with the fullerenes. Tungsten, molybdenum, tantalum, and 304 series stainless steel samples were etched in a nitric acid solution for 1 minute, rinsed and then dried under nitrogen be fore being placed in the ampoules. Only the stainless steel sample was visibly altered after heating to 1073 K. It was coated with a black residue which, when rinsed away, revealed a dulled surface. I'able. 1 is a list of the samples analyzed in this study.

UV-VIS Absorption Analysis

Analysis of the samples was performed by UV absorption spectroscopy using a Cary UV-VIS-NIR spectrophotometer (mode] 5E). A 1.0 cm path length was used in double beam mode with blank solvent in the reference beam path. The powder samples were placed in 200 ml of methylene chloride and sonicated. A further dilution of 1:10 was made for analysis. The concentration of C60 was determined by monitoring the 270 nm absorption feature. The peak intensity was corrected for initial sample weight, then correlated to remaining fullerene material,

Significant quantities of seemingly insoluble material were observed in the CH₂Cl₂ solutions after sonicating for several hours. The volubility of C60 in CH2Cl2 was reported by SRI 25 as 0.26 mg/ml. However, we found that some. of the solid was in fact soluble in toluene. Duc to UV absorption below 250 nJn in toluene, only the 330 nm peak in the C₆₀ absorption spectrum could be used for analysis of tolucne dissolved samples. We prepared two pure C₆₀ solutions - one in toluene, the other in methylene chloride, -10 compare the concentration in each solvent. The concentration of C₆₀ in the methylene chloride, was found to be approximately 10% below that in the tolucne even though identical preparation procedures were used, The concentration of mixed fullerene in the toluene control samples was found to be 77.6 ± 3.5 A cc/mg. The main Sources of error were repeatability of dilution preparation and mass uncertainty.

Results

Figure 4 shows the UV absorption spectrum of mixed fullerene samples heated at 1073 K for various durations. These samples were prepared in methylene chloride.

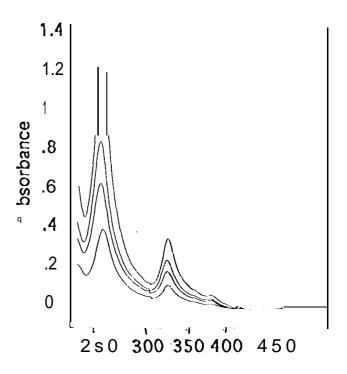


Figure 4, UV absorption spectra of fullerene mix heated for various durations at 1073 K.

Figure 5 shows the degradation rate of the samples mentioned above, toluene dissolved samples with a helium backfill, and samples preheated for 16 hours at 473 K, Note that there is no significant difference in the degradation rates. The samples that had been heated to temperat urcs greater than 1273 K completely decomposed. This toluene insoluble material was examined with x-ray diffraction analysis, The only diffraction peaks to appear were very faint and were attributed to zinc impurities. Though no diffraction pattern from the carbonaceous material could be obtained, the material looked distinctly crystalline. A scanlling-clechron-microscope image of this material is shown below in Figure 7; it dots not appear amorphous. We do not yet have an explanation for the lack of diffraction peaks attributable to this material.

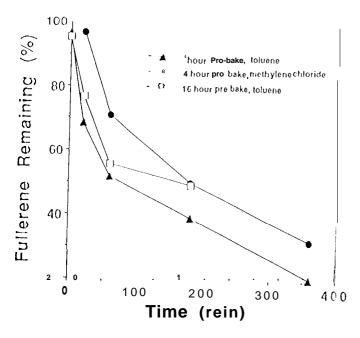


Figure 5. Percentage of fullerene mix reamaining as a function of time. Samples heated to 1073 K.

Figure 6 is a plot of the percentage of toluene soluble material remaining as a function of temperature. Note the apparent stability of the fullerene up to about 973 K.

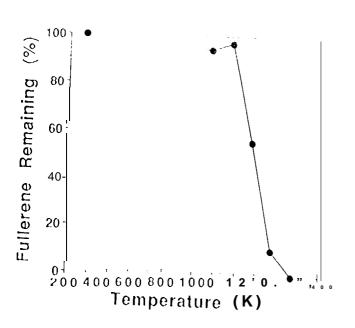


Figure 6. Percentage of fullerenemix remaining as a function of temperature. Samples heated for one hour.

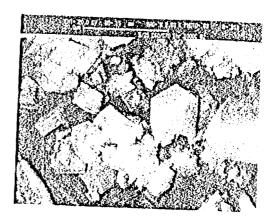


Figure 7. SEM image of insoluble carbon residue from quartz ampoule heated to 1273 K for 1 hour.

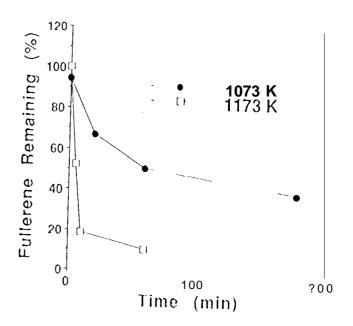


Figure 8. Decay curves for toluene-prepared samples at 1073 K and 1173 K,

By fitting the fullerene decay curves shown in Figure 8, we obtained decay constants, k(T), which were then used to find an Arrhenius activation energy for the disintegration as in the equation,

$$Ln(k) = Ln(A) - \frac{E_a}{RT}$$

where A is the frequency factor (assumed constant here), E_{a} is the activation energy, R is the gas constant, and T is the temperature.

Based on the work reported in References 16-18, and assuming the definition of fragmentation used by Zhang et al., ¹³ we would expect the lower limit of the activation energy 10 be about 40.0 kJ/molfor thermal decomposition if a Lindemann mechanism²⁶ is responsible. In fact, the activation energy for this process is **found to** be **261.5** kJ/mol- a number higher than that predicted by Zhang et al.

Therefore, it is possible that we are observing thermal disintegration of the fullerene mix,

Diffuse Reflectance FTIR Spectroscopic Analysis

Diffuse reflectance FTIR spectroscopy was also used to analyze the decomposed material. Figure 9(a,b,c) shows three spectra: one of untreated full erene mix, a sample healed 10 1073 K for 1 hour, and a sample that had been heated for 18 hours at 523 K in nitrogen and air. Note the, appearance of cyclic anh ydride features in the 1800-1000 cm⁻¹ region of Figure 9c similar to those observed by Vassallo et al. 7 However, no oxidation products are evident in the 1073 K sample, although only 50% of the material remained as full erene. 1 Hydrocarbon impurities are evident in all of the samples. Also, there is a possibility that at these temperatures any intermediate acids will have decomposed, leaving only CO₂ and CO as reaction products and catalysts. We have not attempted to determine whether the sc gases are. present in the scaled ampoules after heating.

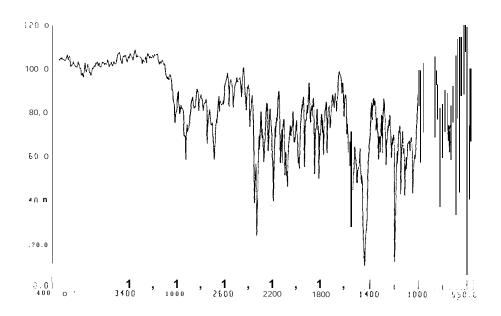


Figure 9a. [4] R Spectrum of C60/C70 untreated

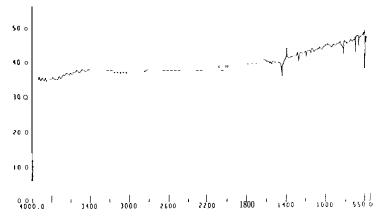


Figure 9b.FTIR Spectrum of C₆₀/C₇₀ heated for 1 hour at 1073 K

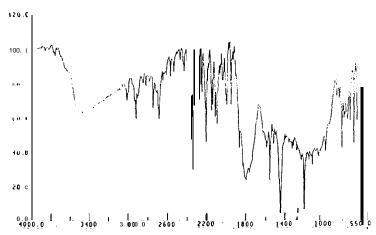


Figure 9c. FTIR Spectrum of C_{60}/C_{70} heated in N₂ and air fo 18 hours at 523 K

Further Experiments

If an RF fullerene plasma discharge is demonstrated to be efficient, a less fragile discharge chamber will be required. A thin silicon carbide or alumina vessel may be a viable. option.

Chamber wall heating may continue to pose a problem. The difficulty with an inductively heated element around the discharge chamber wall is two-fold; l) the wall temperature and RF power will be coupled. Optimized performance of the thruster over a range of operating conditions will then become rnore difficult, and 2) any conducing element will reflect RF power.

The issue of propellant neutralization also has not been adequately addressed, It seems unlikely that hollow cathode neutralizers will be able to operate with a flow of fullerene vapor. Use of a noble gas neutralizer cathode assembly would reduce the system mass benefits associated with using a fullerene ion thruster. 10 a study of the reaction of C60 with tungsten 20, it is noted that because the lowest unoccupied molecular orbital (1 JUMO) of C60 lies deeper than the Fermi level of noble. and transition metals, there is no barrier to charge donation, } lence, negative ion formation of fullerenes

in contact with metals may provide a means for C60 thruster neutral ization.

Conclusions

We have constructed an RF thruster to generate a fullerene plasma and to determine the performance of the system relative to noble gas discharges produced in the same device. We have also investigated the high temperature stability and male rial compatibility of fullerene mix and pure C60 and C70. At temperatures necessary for fullerene ion thruster operation (873 K), C60 remains stable. However, high temperature surfaces such as cathodes pose a problem. We have determined an activation energy of 261.5 kJ/mol for the decomposition of fullerene mix in evacuated quartz ampoules.

Acknowledgments

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